

SIMS Analysis Study of the All Nb SIS Junction

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ABSTRACT. This work presents an experimental investigation of SIS junctions when a thin insulator layer (AlO_x) is introduced between the two superconductor electrodes. The SIMS analysis technique showed the diffusivity effect of the AlO_x as barrier onto the top electrode. The experimental results determine the effect of the AlO_x on the device performances. A general explanation and discussion of this effect has been presented.

Introduction

Secondary Ion Mass Spectrometry (SIMS) is the technique that has the widest range of configurations. SIMS instrumentation ranges from the “add on” quadrupole to the top of the range dedicated dynamic SIMS instrument^[1].

Basic Principles

The basic principle behind SIMS is the sputtering process which is also used for removing surface layers in the other surface analysis techniques. The sputtering process is shown schematically in (Fig. 1). The primary ion beam is directed at the sample surface and the primary ions penetrate the solid surface colliding with the atoms in the sample and losing energy. Eventually the primary ions come to rest some distance below the sample surface, the initial energy of the primary ion being transferred to the atoms in the sample displacing them from their initial positions. The displaced atoms in turn collide with other atoms producing a collision cascade. Sufficient energy is transferred by the collision cascade to atoms in the immediate surface that some of them can be ejected into the vacuum.

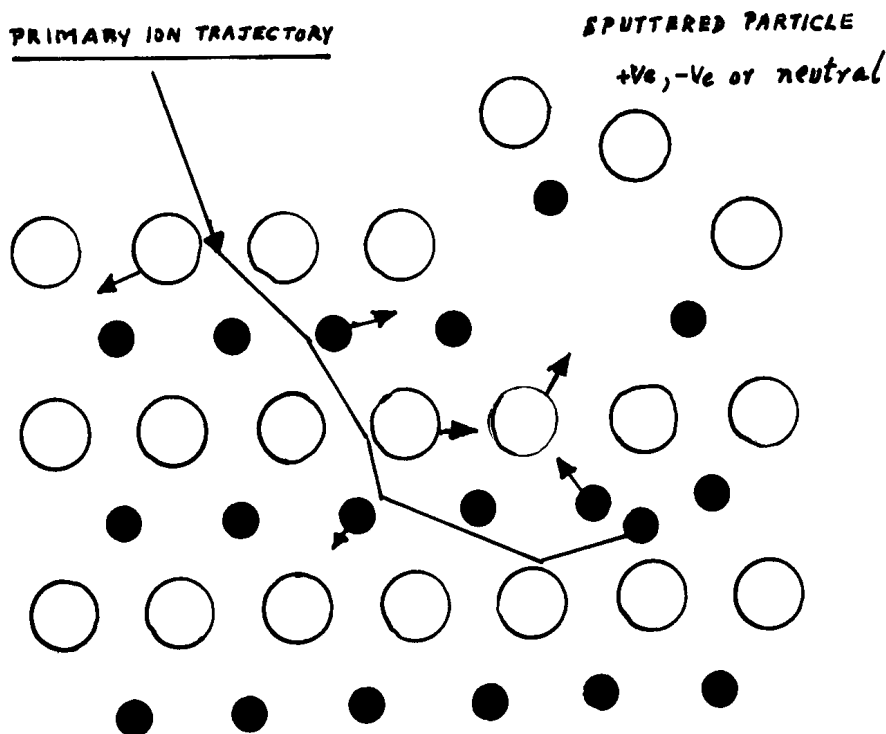


FIG. 1. Schematic diagram of the sputtering process.

The primary ion implantation depth and the collision cascade can extend for a significant distance into the sample surface depending upon the primary ion energy. However, only those atoms in the immediate surface layer have a significant probability of being ejected. SIMS is therefore a surface technique with the information coming principally from the outer monolayer although that surface moves into the sample in the course of the analysis.

The sputtered particles emitted from the surface can be single atoms or molecules in neutral, excited or ionized states, either positively or negatively charged. As far as the other surface techniques are concerned it is of little consequence in what form the atoms are sputtered from the surface but for SIMS it is only the ionized component that can be detected. Argon ion bombardment could be used for SIMS, and indeed is for static SIMS, but it is found in practice that the number of secondary ions produced is critically dependent on the local pressure of oxygen in the region of the sample surface.

By using a primary ion beam of oxygen ions rather than argon ions, this effect can be exploited to increase the number of secondary ions produced. The use of an oxygen primary beam saturates the sample surface with oxygen and maximizes the ion yields, at least for positive secondary ions from electro-positive elements.

The basic components of any SIMS system are the same for static or dynamic SIMS: a primary ion source, a mass spectrometer, a vacuum system and a data acquisition system.

The ion guns used for dynamic SIMS are designed to operate in the 1 keV to 20 keV range delivering currents of several microamps into spots a few microns in diameter producing etch rates of tens of Angstroms per second, whilst for static SIMS lower energy less focused beams are used to give etch rates of the order of Angstroms per hour.

The secondary ion current, I_m^+ , arriving at the detector is given by^[1]:

$$I_m^+ = J_p \cdot A \cdot S \cdot \beta_m^+ \cdot f \cdot C_m$$

where J_p is the primary ion current density

A is the sample area

S is the sputter yield

β_m^+ is the degree of ionisation

f is the instrument transfer function, and

C_m is the concentration of the element

To have the highest sensitivity, it is necessary to maximize I_m^+ . This can be achieved by increasing the sampled area, the primary current density, and the sputter yield.

Two types of mass spectrometer are used for SIMS analysis: the quadrupole and the magnetic sector. The quadrupole is used for static SIMS and some dynamic SIMS instruments, whilst the magnetic sector mass spectrometer is found only in dedicated dynamic SIMS instruments.

The quadrupole, as its name suggests, is composed of four rods to which a combination of d.c. and r.f. voltages are applied. Ions of a particular charge to mass ratio can follow stable trajectories between the rods depending upon the value of the applied fields. By varying the applied field, ions of different charge to mass ratio can pass between the rods and reach the detector. Quadrupoles offer high mass ranges and compatibility with conventional u.h.v systems, but suffer from limited mass resolution and low transmission^[2].

In the magnetic sector mass spectrometer, the ions are accelerated by an electric field and then injected into a magnetic field in which they follow circular orbits with radii determined by their charge to mass ratio.

Figure (2) shows the instrument used, a CAMECA 1ms 3f dynamic SIMS system equipped with a duoplasmatron ion source (for O_2^+ and O^- ions) and a C_s^+ ion source together with a mass filter to ensure beam purity. The instrument employs a double focusing magnetic sector mass spectrometer with variable mass resolution (250 to 10,000) and a mass range of 0 to 250 amu. Analysis can be performed on areas from 2 μm in diameter to 400 μm in diameter and is capable of sub-micron resolution in the ion imaging mode. The instrument is computer controlled and offers the following modes of operation^[3,4]:

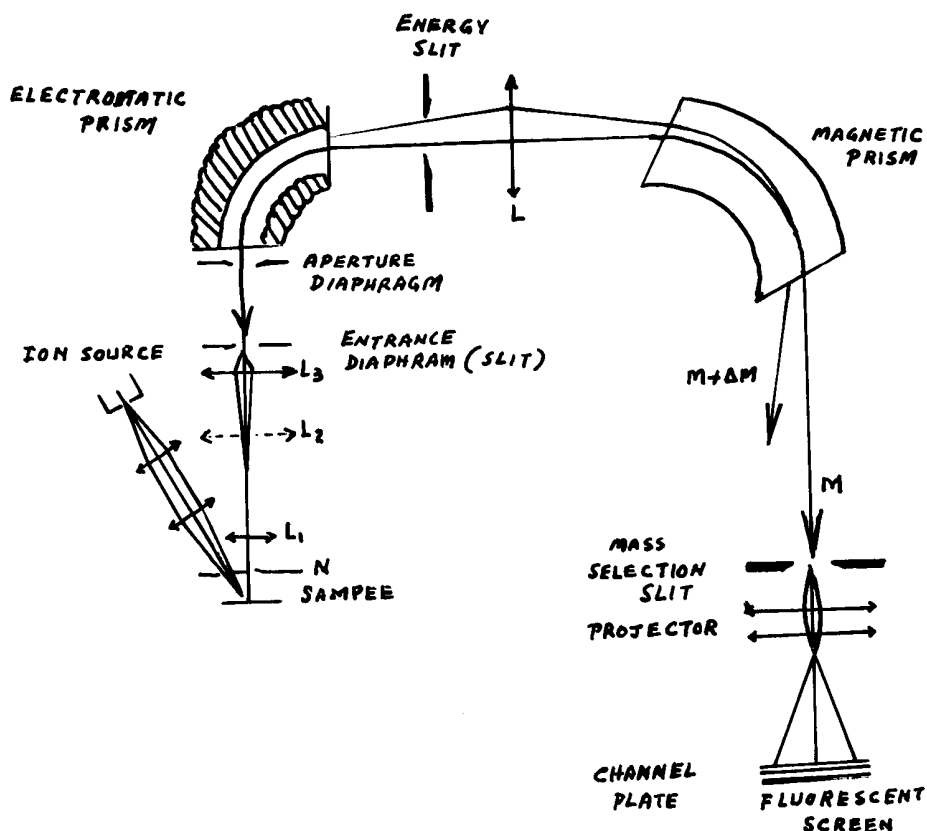


FIG. 2. Schematic diagram of the cameca IMS 3F instrument.

Barograph mass spectra over the entire mass range
Continuous mass spectra over a limited mass range
High resolution mass spectra of individual mass peaks
Ion energy distribution measurement
Step scanning across the sample in any direction to determine lateral variations in composition.
Depth profiling to determine the variations in compositions with depth
Isotope ratio measurements.

Experimental Technique

The starting material is "MARZ" grade niobium of 99.999% purity in 3×12 mm disc form. The other material used was aluminum wire of 99.999% purity. The aluminum film thickness at this stage is very important for the construction of the Nb-AlO_x-Nb junctions.

Prior to the degassing as well as evaporation procedures, the UHV system was evacuated to 10^{-8} mbar, then baked for several hours with a takeout temperature of 200-250°C.

This treatment provides a chamber base pressure of 10^{-9} mbar. Then, the niobium disc was degassed several times. When its temperature was raised near its melting point, the pressure rose to 10^{-7} mbar. These degassing procedures were done successively until the pressure gradually returned to 10^{-9} mbar with degassing still running. At this vacuum the cleaning period was ended and the titanium sublimation pump connected the system with the main chamber cold shroud held at 77°K by filling with liquid nitrogen. At this stage, the pressure falls to below 10^{-10} mbar.

The mass spectrum was normally taken in three separate periods, i.e. one before starting the evaporation, in the middle, and after the end of the evaporation.

A typical spectrum of the residual gases as determined by the mass analyser is shown in Figure (3). The atomic mass identifies the particular element or compound present, whereas the peak height gives an approximate measure of the partial pressure. Under this condition the first Nb electrode was deposited.

By the end of evaporation of this first electrode substrate carrier was moved to the oxidation chamber and the gate valve was closed to secure the cleanliness of the main chamber. Then aluminum film was deposited immediately onto the top of the first niobium electrode under appropriate angle.

Pure oxygen was thus leaked to the oxidation chamber in order to produce an insulator layer in the form of AlO_x. The next step was the removal of oxygen from the oxidation chamber using diffusion pump, then the gate between the two chambers was opened, and when the pressure returned to 10^{-9} mbar the

second Nb electrode was deposited. Then the sample was moved to another UHV system for SIMS analysis.

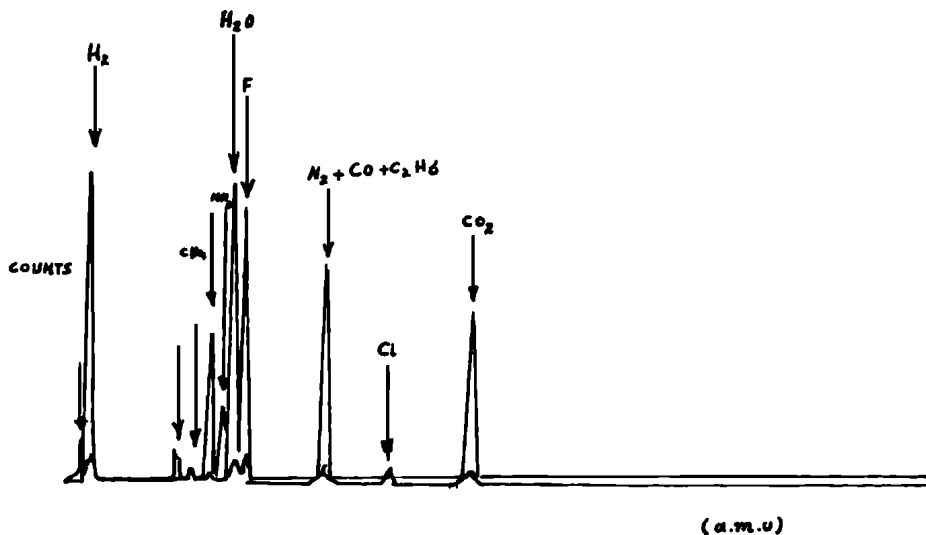


FIG. 3. Typical spectrum of the residual gases inside the chamber before the SIMS analysis started.

Results and Discussion

The results of SIMS analysis appear firstly in a form of mass spectrum of which niobium and aluminum peaks are of primary importance, because of the assumed Nb-AlO_x-Nb form.

Initially mass scans were carried out and one set of results are shown in Fig. (4) taken at 15 keV excitation energies for Nb-AlO_x-Nb surfaces.

Figure (4) shows the mass analysis of the ions produced by a douplasmatron source. The x-axis represents the atomic mass number (a.m.u), while the y-axis gives the number of counts. The spectrum of the sample with the highest Nb purity is shown for comparison of the Nb line with relative intensity of niobium-oxide and niobium-dioxide lines. The atomic purity of the extracted beam under these conditions is good, while the main role of the magnetic sector is to analyze them into ions components.

From the spectra, it can be seen that niobium and aluminum exist in Nb-AlO_x-Nb samples. Clearly this method provides a means of detailed assessment of film composition and the element distribution of their chemical states throughout the film. Because this technique is very sensitive, it is easy to indicate even small amounts of impurities distributed all over the niobium films^[5].

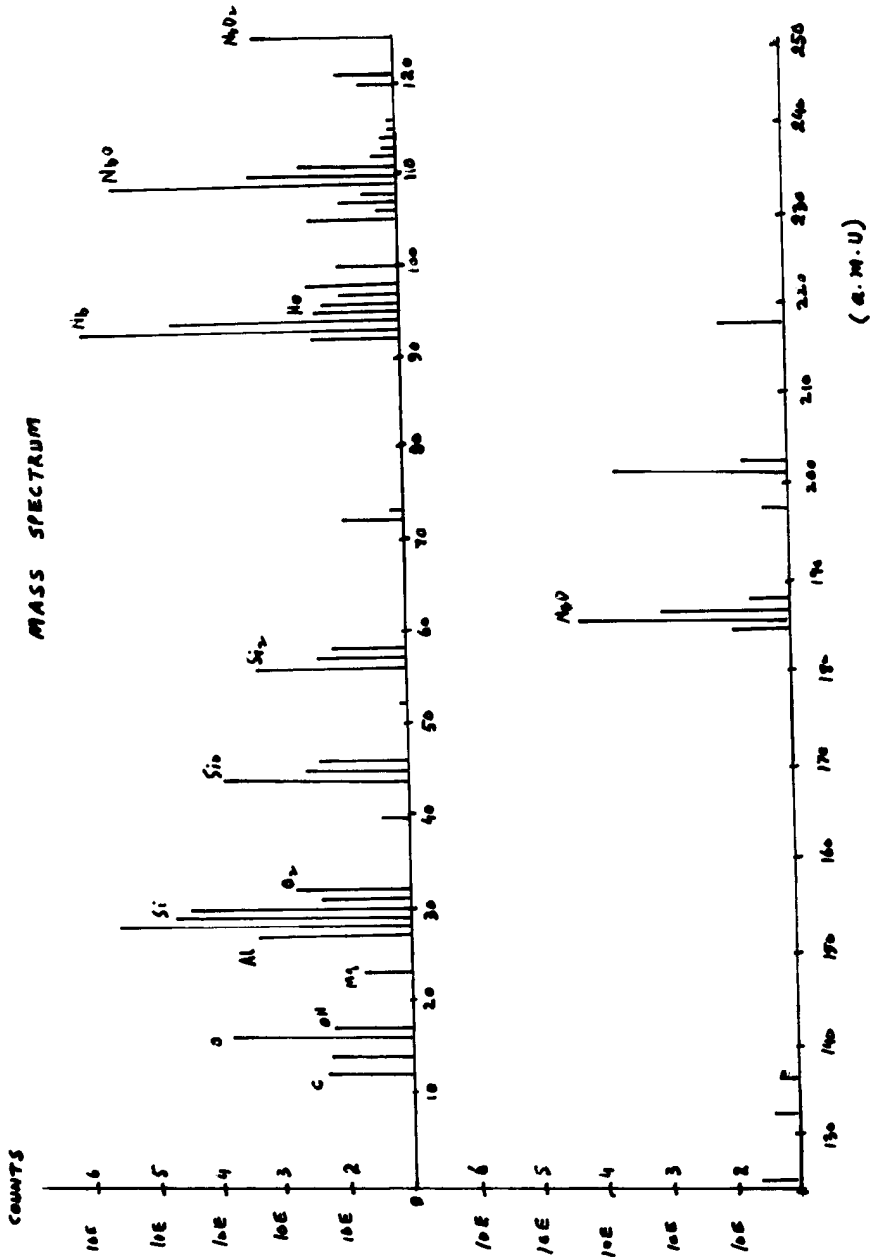


Fig. 4. Typical mass spectrum for Nb-AlO_x-Nb junction.

When the samples were analyzed using an O_2^+ primary source, the resulting SIMS spectrum revealed significant quantities of Mo (95,98 a.m.u), and Si (30 a.m.u) as shown in Fig. (5).

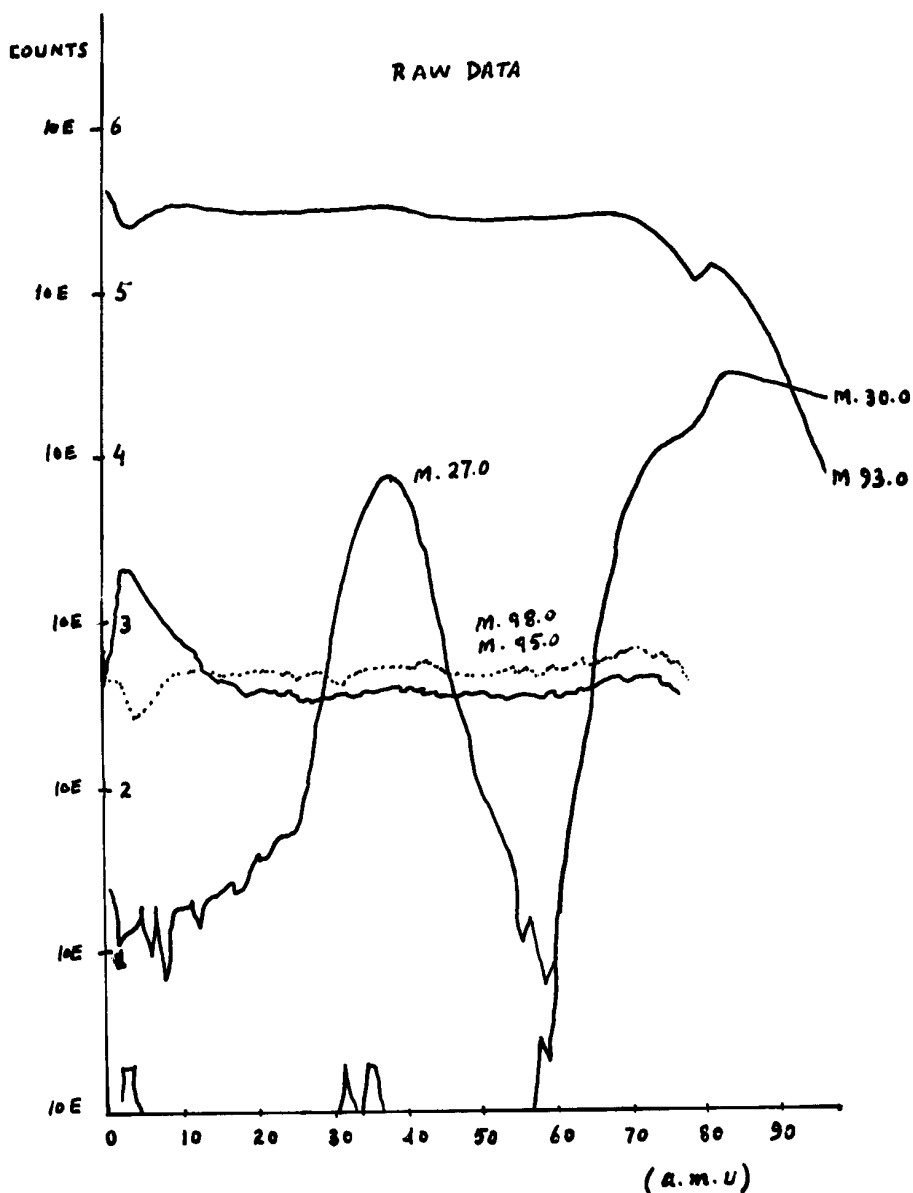


FIG. 5. Depth profile of SIMS for Nb- AlO_x -Nb sample using O_2^+ bombardment.

However, with a further ion milling for depth profiling the SIMS signal has shown a discontinuity exhibited by sudden appearance of a Si signal, obviously corresponding to the substrate. By the successive appearance of signals from Nb, Al, and Nb, it is confirmed that the junction has the desired composition^[6,7].

Analysis was also performed using C_s^+ bombardment and negative ion detection in order to optimize the sensitivity for C and O.

Isotope O^{18} was monitored rather than O^{16} due to the oxygen levels being very high as in Fig. (6). The C and O levels were observed to follow the aluminum profile in the Nb- AlO_x -Nb sample. The profiles in these two figures exhibit clearly a high and uniformly distributed amount of both elements. The Mo peaks appear to be constant throughout the film at a level of 0.1%, but the Na was variable. In Fig. (6), the carbon appeared to start with high level, and steadily dropped to its lower value at the substrate surface region.

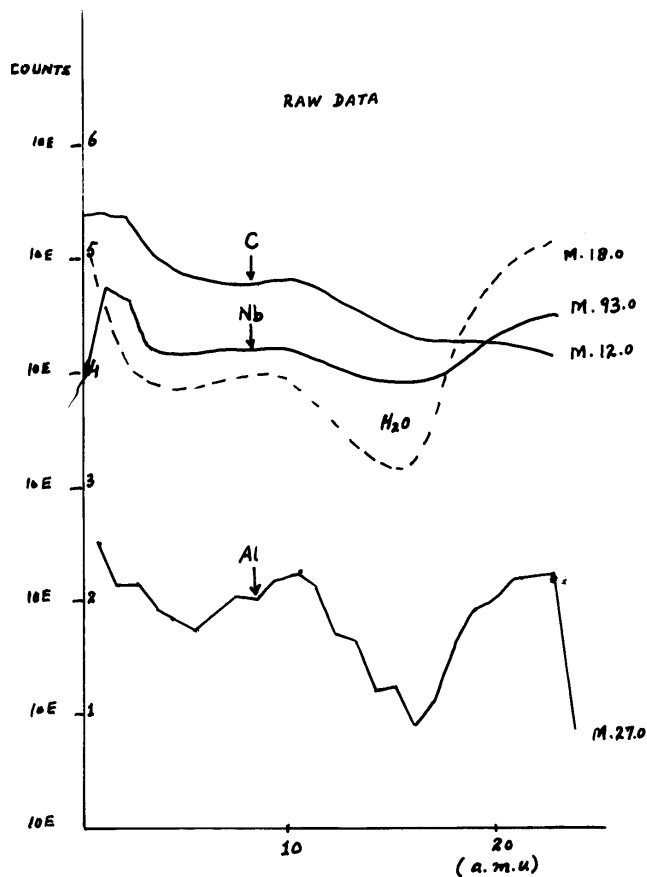


FIG. 6. Depth profile of SIMS Nb- AlO_x -Nb sample using C_s^+ bombardment.

Oxygen appeared at a very high concentration at the surface of the film and dropped to at least 10% at the middle region. It showed its highest level for regions near the substrate surface.

In sample Nb-AlO_x-Nb indicated in Fig. (5), the Mo kept at level 0.1% throughout the film, aluminum starts at low level and reaches its maximum peak at 1.7% around the middle region and then dropped to zero at the substrate surface.

Molybdenum appears clearly in both samples at a very low level of 0.1% and that may be attributed to e-gun hearth which is made from this material, while oxygen may be due to the residual gasses in the UHV system during deposition^[8]. It is difficult to be specific about the source of carbon or sodium that may result from contamination subsequent to removal from the deposition chamber. Further, some of the signals could be induced from SIMS analysis itself^[9,10].

It is not possible to detect the interface layer between the two films by SIMS techniques indicating that contamination in the deposition system is at an acceptably low level.

Conclusion

Several All Nb SIS Junctions have been prepared by using the e-beam evaporator. These junctions were examined by SIMS techniques. In this techniques, the O₂⁺ bombardment and positive ion detection were used in order to optimize the sensitivity of Nb and Al. The sample Nb/AlO_x/Nb showed uniform contamination by Mo. The Al profile showed evidence of migration of Al onto the top layer of Nb. For the same sample C_S²⁻ bombardment and negative ion detection were used and it was observed that the C and O levels were very high, especially the O levels. However inspite of the diffusivity effect on to the top Nb electrode, the device quality was not seriously affected^[11].

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دراسة تحليلية لموصل كهربائي بواسطة جهاز SIMS

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المستخلص . يقدم هذا البحث دراسة تجريبية لموصل كهربائي مصنّع من المواد الفائقة التوصيل مكون من قطبين مفصولين عن بعضهما بعازل، وأجريت دراسة على هذه الأقطاب بواسطة جهاز SIMS . ونتيجة لذلك تبين أن هناك انتشاراً للمادة العازلة إلى القطب العلوي، وقد أوضحت هذه الدراسة تأثير ذلك الانتشار على هذه الأنواع من الموصلات الكهربائية .